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Abstract
A compact system is developed to measure laser absorptivity for a variety of powder materials (metals, ceramics, etc.) with different powder size distributions and thicknesses. The measured results for several metal powders are presented. The results are consistent with those from ray tracing calculations.

There exists a need for the measurement of powder absorptivity for the different variants of the additive manufacturing (AM) processes.
In addition to a dependence on wavelength and intrinsic material parameters, the laser absorptivity of powder depends on the particle shape, size distribution, porosity, and thickness of the powder layers.
As a result, there is increasing demand for a simple compact system for fast measurements of the temperature-dependence of the laser absorptivity up to and including the molten state. Existing systems, e.g. [1], measure the reflected light from the powder with the help of an integrating sphere and are typically complex and expensive. The distribution of the scattered light is broad and even the small absorption in the integrating sphere coating can affect the result.
We propose a simple calorimetric scheme for direct absorptivity measurements. The scheme of the measurements is presented in Fig.1. A thin layer of powder is placed on a thin disk made from refractory metal. A laser or diode array beam uniformly irradiates the thermally isolated disk. The temperature increase is measured by thermocouples underneath the disk. The disk holder is designed such that it does not significantly absorb radiation nor affect the temperature distribution in the target. The input heating is selected to be slow compared to the rate of thermal diffusion, resulting in a uniform temperature through the powder and substrate. The temperature across the face of the disk will be consistent due to the uniform nature of the laser irradiation.

Consider a thin layer of powder with thickness d_1 on a flat disk of refractory metal with thickness d_2 and radius R uniformly illuminated by light with intensity *I*. For absorptivity of powder (or melt), assuming uniform temperature through the disk, the temperature evolution is:

$$\left(\rho_1 c_1 d_1 + \rho_2 c_2 d_2\right)\frac{dT}{dt} = A(T)I - Q(T) \tag{1}$$

where *A(T)* is the absorptivity, *Q(T)* the thermal losses including convective and radiative, ρ the density, *c* the specific heat, *d* the thickness, and subscript "1" for powder and "2" the substrate.

Consider a flat top, finite duration heating pulse. A typical temperature history is presented in Fig. 2 and comprises two phases, heating and cooling. First we consider the temperature evolution during the cooling phase, when $I = 0$ in order to determine the convective and radiative losses Q(T) for known heat capacities and material densities. Next we will be able to find from (1) the temperature dependent absorptivity considering the temperature evolution during the heating phase. The missing piece in this scheme is the measurement of the powder density (porosity). We solved this problem with a special target design. The target disk with diameter d has a rim (Fig.3) with height h to determine the powder thickness. The disk is filled with powder and a blade or roller removes the extra material, mimicking the powder deposition of a commercially available AM system. If we multiply Eq. (1) by the disk area S, the equation can be rewritten as

$$\left(m_1 c_1 + m_2 c_2\right)\frac{dT}{dt} = A(T)P - Q(T)S \tag{2}$$

Here m_1 and m_2 are the masses of the powder and disk, respectively. *P=IS* represents the total power incident on disk.
Weighing the disk with powder and without gives the powder weight needed to calculate the absorptivity from Eq. 2. A similar set-up was used in a previous study to measure the absorptivity of solid metal, where more details of physical effects related to the experiment can be found [2]. We also used a data processing algorithm developed in that work.

The specific set-up used in the experiments is presented in Fig. 4. Emission from three vertical-cavity surface-emitting lasers (VCSEL) radiating at 970 nm were imaged and angularly beam combined onto a tantalum disk with a prepared powder layer. The wavelength was selected close to the 1 μm radiation used in typical AM systems. The emission profile is relatively flat over the 1 cm diameter aperture of the sample disk, providing the desired uniform thermal distribution. The tantalum disk with several K-type thermocouples attached underneath is carefully placed onto two thin metal wires with diameters between 0.003 and 0.01 inches. The wires are held tautly on a translation stage and provide stability and positioning adjustability for the sample disk while minimizing heat conduction. Estimates show that heat flux through the wires is small and can be disregarded. Temperature histories from the various thermocouple locations were identical, supporting the assumption of uniform temperature across the disk.

The specific measurements were done for 316 stainless steel, two Ti-6Al-4V alloys and a 99.9% purity Al powder. The density and heat capacity of Ta as function of temperature were taken from [3]. For stainless steel, Ti alloy and Al, we used the density and heat capacities from [4].

The results of the measurements are presented in Fig.5 (a, b, c). The stainless steel powder (Fig. 5a) was the same as used in experiments carried out in a Concept Laser AM machine [5]. The powder has a Gaussian size distribution with average radius 13.5 μm, a full width at half maximum of 2.3 μm, radial cutoffs at 8.5 μm and 21.5 μm.[5] After the first measurement the sample was allowed to cool and the measurement was repeated (blue lines). We observed some small difference in results, probably due to powder re-configuration driven by thermal expansion. Performing the measurement at two laser intensities gave consistent results, suggesting that the absorptivity is independent of the heating rate.

We used Ti-6Al-4V powder from two different suppliers. The powders have different particle size distributions (with the same average diameter~27 μm, same as for stainless steel) and behaved differently when spread across the target disk. One powder was more cohesive than the other, tending to stick to the coater blade and roller and to form clusters. We used a glass plate to create a smooth flat powder surface. Interestingly, the measured absorptivity was insensitive to the powder suppliers.

The measurements presented here were done at temperatures up to 500°C. At higher temperatures, oxidation becomes important and the material changes color [2]. In a typical additive manufacturing process the melting takes place in an Ar environment and we plan to make our high temperature measurements under similar conditions. The use of a Ta disk gives a possibility to go above the melting points of most materials of interest and measure the absorptivity of the melt.

Let us compare our results with recent, first principles modeling of laser absorption in powder using a ray tracing code. [6]. It was demonstrated that due to multiple scattering the powder absorptivity is greatly increased in comparison to flat surface absorptivity. The absorption for the metals with high absorptivity (SS, Ti) is practically independent of powder structure. For stainless steel, the calculations in [6] give 60% absorptivity for the monosized hexagonally packed powder and 58% for powder with experimentally measured size distributions packed according to the "rain drop" method [7]. Experimental measurements are consistent with these calculations.
The insensitivity of absorption to the powder structure may explain the independence of Ti-6Al-4V absorption on powder type. The absorptivity value for Ti alloy in our measurements is about 70%, somewhat higher than predicted by the modeling value ~65% [6]. One possible explanation is that the calculations in [6] used the refractive index for the pure Ti, which can differ from that of Ti-6Al-4V.
Calculated values for Al are very different from our measurements. It suggests that the oxide layer and the structure of the surface are important. For a flat surface, the observed absorptivity of Al is over 20% for 1 μm light, much higher than the 5% value predicted using the textbook refractive index (see discussion in [2]). The increase in powder absorptivity in comparison with a solid material is consistent with numerical results [6].

We developed simple and compact set-up for the direct measurement of the temperature-dependent powder absorptivity. The results for three metals are presented. The results are in good agreement with the direct numerical calculations except for Al, which can be explained by the effect of the oxide layer.

Acknowledgments

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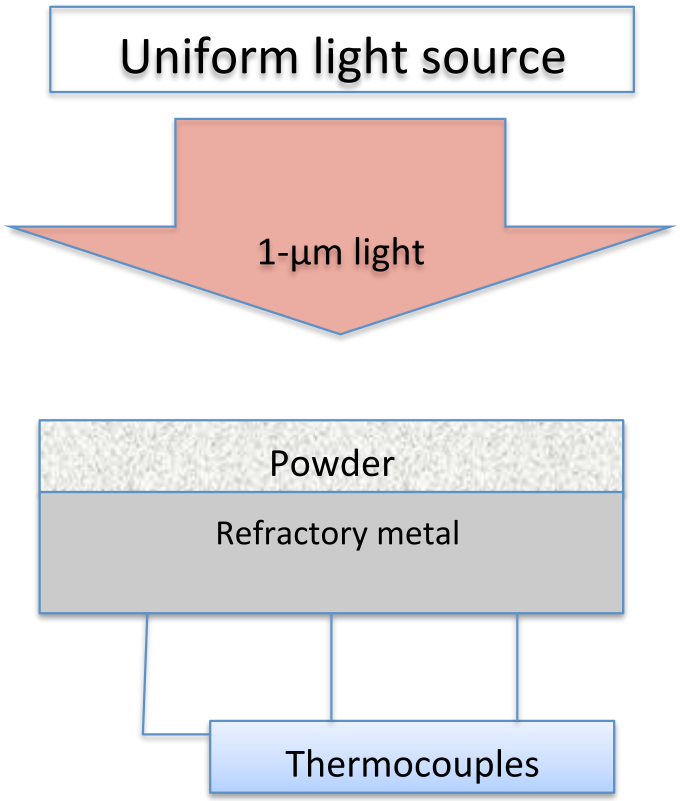


Fig 1. Diagram of the measurement scheme. A thin layer of powder is placed on a thin disk made from refractory metal and is uniformly irradiated by 1-µm laser light sources. Temperature is measured by thermocouples attached to the bottom of the disk.

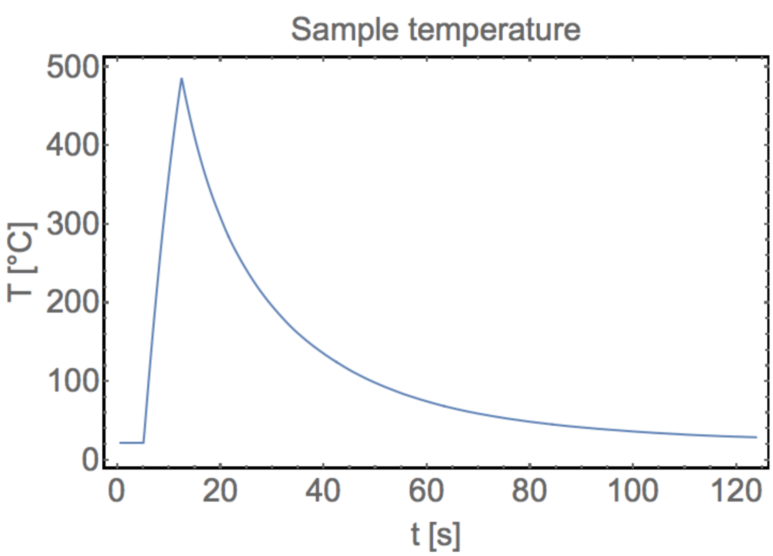


Fig. 2. Sample data from thermocouples attached to the refractory disk showing the temperature variation during the heating and cooling periods.

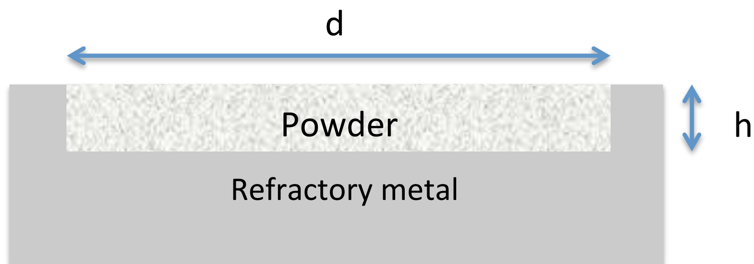


Fig. 3. The target disk of diameter d has a rim of height h. The disk is filled with powder and a blade or roller removes the extra material, mimicking the powder deposition in a commercially available AM system.

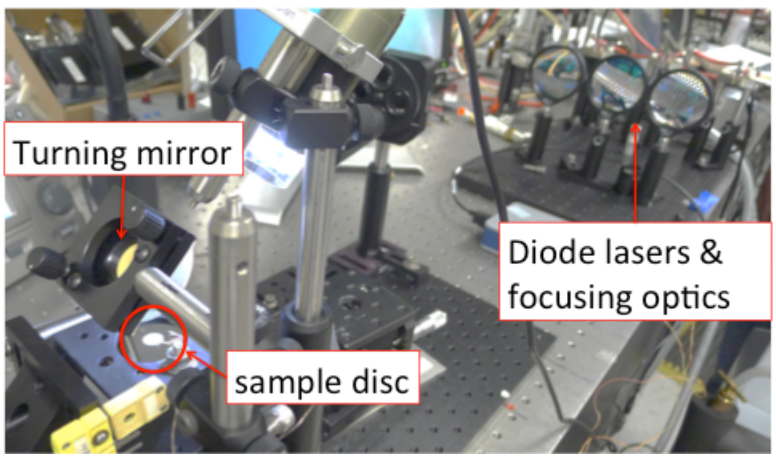
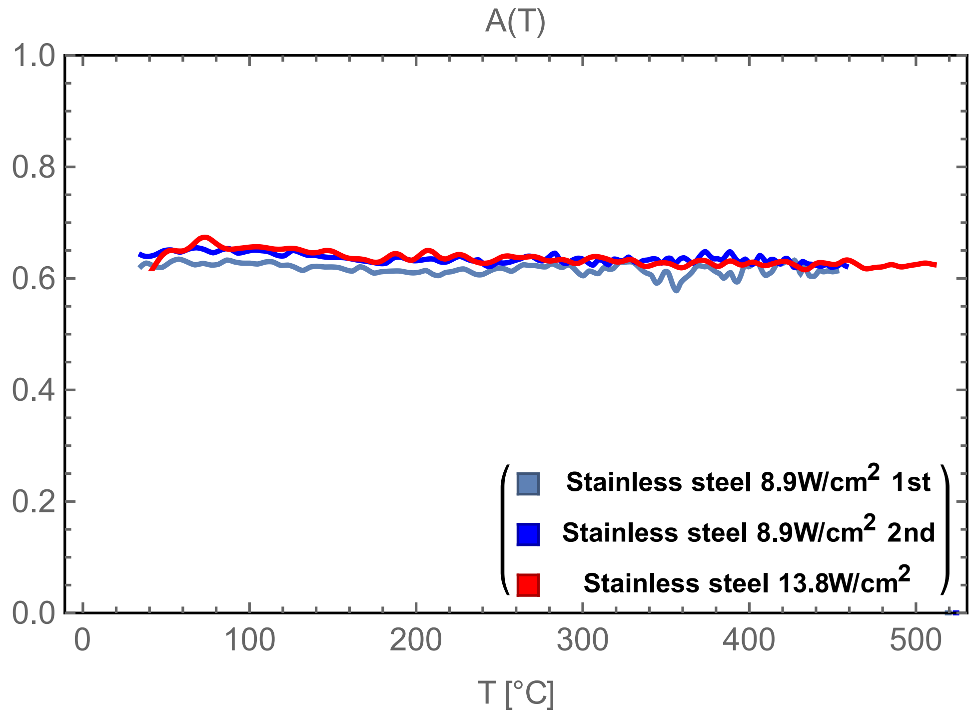
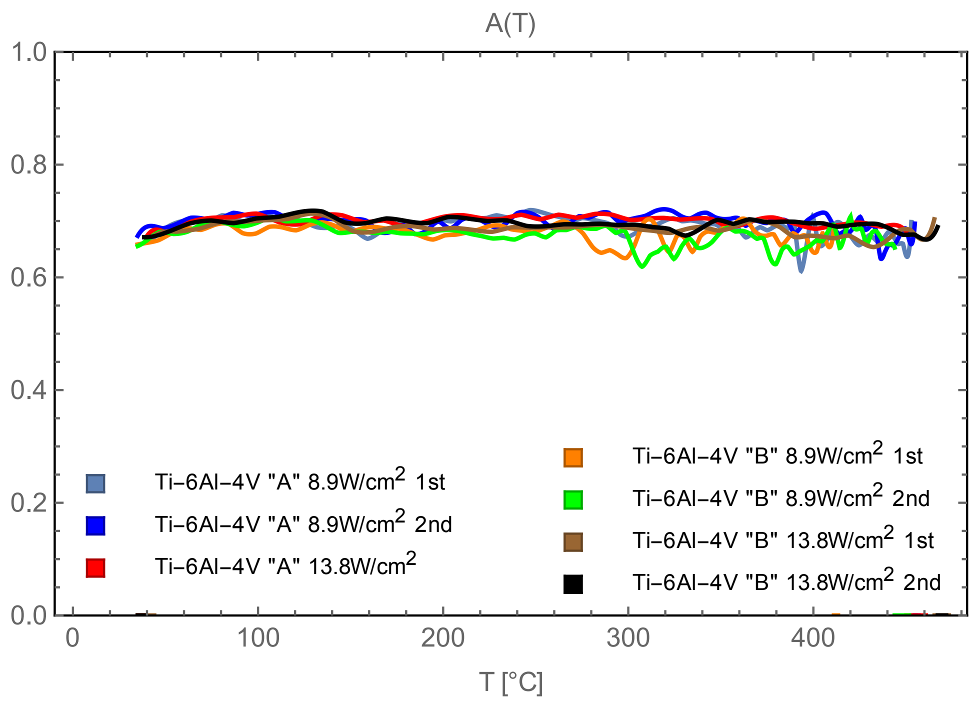


Fig 4. Output from three vertical-cavity surface-emitting lasers (VCSEL) are imaged and angularly beam combined onto the target disk. The target disk with thermocouples attached underneath is carefully placed onto two thin positioning wires stretched over a translation stage.



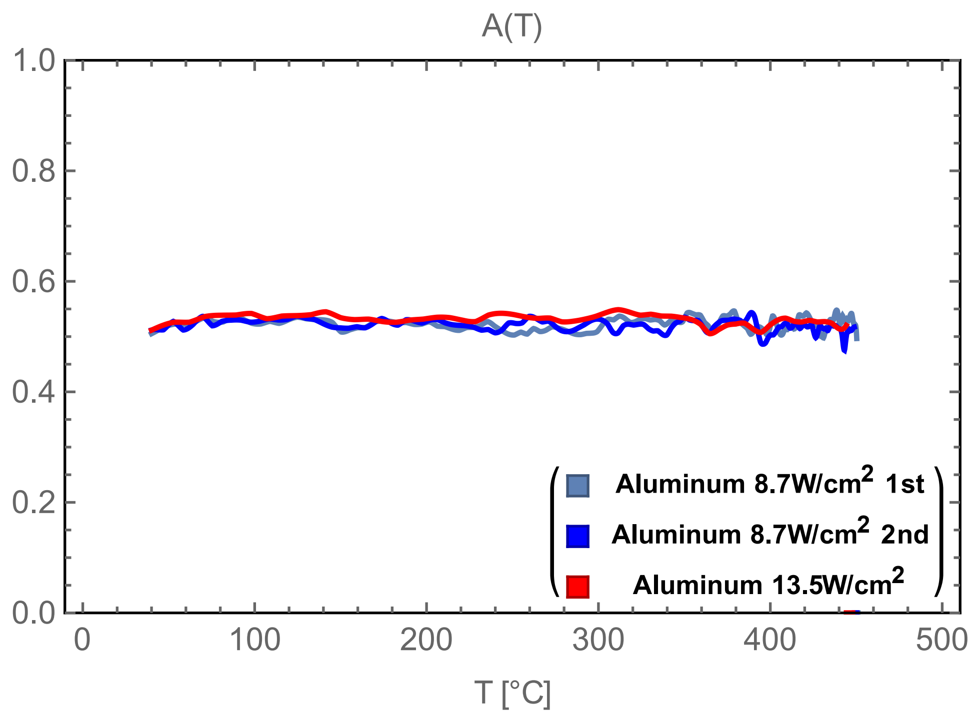
A(T)
1.0
0.8
0.6
0.4
0.2
0.0
0
100
200
300
400
500
T [°C]
Stainless steel 8.9W/cm2 1st
Stainless steel 8.9W/cm2 2nd
Stainless steel 13.8W/cm2

(a)



A(T)
1.0
0.8
0.6
0.4
0.2
0.0
0
100
200
300
400
T [°C]
Ti–6Al–4V "A" 8.9W/cm2 1st
Ti–6Al–4V "A" 8.9W/cm2 2nd
Ti–6Al–4V "A" 13.8W/cm2
Ti–6Al–4V "B" 8.9W/cm2 1st
Ti–6Al–4V "B" 8.9W/cm2 2nd
Ti–6Al–4V "B" 13.8W/cm2 1st
Ti–6Al–4V "B" 13.8W/cm2 2nd

(b)



(c)

Fig 5. Measured absorptivity data for (a) stainless steel 316, (b) Ti-6Al-4V, and (c) 99.9% purity Al (Goodfellow Al006031).